(12) INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(19) World Intellectual Property Organization
International Bureau

(43) International Publication Date
4 January 2001 (04.01.2001)

PCT

(10) International Publication Number
WO 01/00048 A1

(51) International Patent Classification[7]: A23L 2/44, A23F 3/16, A23L 3/3553, 2/02

(21) International Application Number: PCT/US00/17548

(22) International Filing Date: 26 June 2000 (26.06.2000)

(25) Filing Language: English

(26) Publication Language: English

(30) Priority Data:
60/141,295 28 June 1999 (28.06.1999) US

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(81) Designated States *(national)*: AE, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CR, CU, CZ, DE, DK, DM, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, TZ, UA, UG, US, UZ, VN, YU, ZA, ZW.

(84) Designated States *(regional)*: ARIPO patent (GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG).

Published:
— *With international search report.*
— *Before the expiration of the time limit for amending the claims and to be republished in the event of receipt of amendments.*

For two-letter codes and other abbreviations, refer to the "Guidance Notes on Codes and Abbreviations" appearing at the beginning of each regular issue of the PCT Gazette.

WO 01/00048 A1

(54) Title: NON-CARBONATED BEVERAGES COMPRISING ANTIMICROBIAL LONG CHAIN POLYPHOSPHATES

(57) Abstract: Polyphosphates for use in beverages and methods for their preparation. Long chain polyphosphate in which the number of phosphate units is greater than 22 are superior materials for the control of yeast and mold growth in beverages so that the level of preservatives in the beverage can be reduced, minimizing or eliminating the undesirable flavor associated with high levels of preservatives. Use of polyphosphates in which n is greater than about 45 is advantageous. Use of polyphosphates in which n is greater than about 75 is especially advantageous.

NON-CARBONATED BEVERAGES COMPRISING ANTIMICROBIAL LONG CHAIN POLYPHOSPHATES

FIELD OF THE INVENTION

This invention relates to polyphosphates useful in beverages. In particular, this invention relates to long chain polyphosphates, which are effective against yeast and mold growth.

BACKGROUND OF THE INVENTION

Many beverages provide an excellent environment for rapid microbial growth because microorganisms can rapidly proliferate by feeding on nutrients present in the beverage. Fruit juice for example, an important component of many beverages, provides an excellent medium for the growth of microorganisms, especially yeasts and molds. Consequently, controlling microbial growth in packaged beverages, especially those that are stored under ambient conditions, is an ongoing concern among beverage manufacturers.

Although beverages can be maintained under ambient conditions if they are heat pasteurized during packaging (hot packing) or are packaged under completely aseptic conditions (aseptic packaging), not all beverages can packaged by these methods. Hot packing, which involves heating the beverage and its container to a temperature between about 85-105°C during packaging so that the resulting sealed beverage contains no food spoilage microorganism, is unsuitable for manufacturing certain types of beverages. Hot packaging is commonly used in the manufacture of beverages that are canned or bottled in glass containers, but flexible containers made from high density polyethylene, for example, cannot be subjected to the temperatures used during hot packing operations. Although packaging under completely aseptic conditions produces a beverage free of food spoilage microorganisms, aseptic packaging methods are often unsuitable for manufacturing beverages packaged in certain beverage containers, *e.g.*, rigid containers such as glass and cans. In addition, a sterile environment is difficult to maintain during the packaging operation. Frequent cleaning of the packaging line, which is time consuming, expensive, and frequently ineffective in preventing microbial contamination, is necessary.

Because these methods cannot be used for all types of beverages and because these methods sometimes do not produce a beverage that is free of microorganisms, preservatives, such as sorbates, benzoates, and organic acids, are often added to inhibit microbial proliferation. However, when used at the levels necessary to inhibit subsequent microbial proliferation at ambient temperatures, preservatives often contribute an off-flavor to the

beverages. But, when used at concentrations sufficiently low to avoid an off-flavor, preservatives are generally unable to effectively inhibit the growth of many food spoilage microorganisms.

Sodium salts of phosphoric acid derivatives, especially food grade sodium hexametaphosphate, have been used with preservatives to enhance the potency of preservatives so that lower levels can be used, thus improving taste. Calderas, U.S. Pat. No. 5,431,940, incorporated herein by reference, for example, discloses the use of polyphosphates in combination with sorbate preservatives in dilutes juice beverages having relatively low water hardness.

However, in most cases, phosphate salts cannot completely replace preservatives, so preservative taste may still be required to produce the desired product stability. Thus, a need exists for phosphate salts that are more effective than simple phosphates in controlling microorganisms, especially yeasts and molds.

SUMMARY OF THE INVENTION

In one embodiment the invention is a beverage containing a long chain polyphosphate in which the average number of phosphate units is greater than 22. The long chain polyphosphate controls yeast and mold growth. This extends the shelf life of the beverage and allows the level of preservatives in the beverage to be reduced, minimizing or eliminating the undesirable flavor associated with high levels of preservatives.

DETAILED DESCRIPTION OF THE INVENTION

Long Chain Polyphosphates

Long chain polyphosphates control the growth of different classes of microorganisms that could lead to spoilage in fruit-flavored or flavored non-carbonated beverages having a pH of less than about 4.5, preferably less than or equal to 4.0. It has been discovered that long chain polyphosphates in which n, the number of phosphate units is greater than 21, are more effective than potassium sorbate, sodium benzoate, or a commercially available food grade amorphous, water soluble sodium polyphosphate composed of linear chains of metaphosphate units with an average chain length of 13 against yeasts, based on a comparison of their minimum inhibitory concentration values (minimum concentration that inhibits growth of the microorganism). These materials are also more effective against molds than shorter chain polyphosphates.

Long chain polyphosphates have by the following structure:

$$M_{(n+2)}O(PO_3)_n$$

in which n is the average number of phosphate units in the polyphosphate and M represents a metal cation, typically sodium ion or a mixture of sodium and potassium ions. Long chain polyphosphates in which n is greater than 22 are more effective than shorter chain materials. Preferably, n is greater than or equal to 28, more preferably greater than or equal to 45, and even more preferably greater than or equal to 75. n may be as large as 100 and may be as large as desired, provided the polyphosphate has the requisite solubility for beverage applications and the solutions do not become so viscous that they cannot be conveniently used. A 1% solution preferably has a pH of 2.0 to 4.0.

Long chain polyphosphates may be prepared by heating a phosphate salt, such as NaH_2PO_4, at about 750°C to drive off water and to form a clear melt. Heating below 600°C produces materials with insufficient chain length.

Beverages

The preparation of beverages and the materials used therein is well known to those skilled in the art and has been described in numerous patents and publications, such as, Nakel, U.S. Pat. No. 4,737,375, which emphasizes the preparation of carbonated beverages; Calderas, U.S. Pat. No. 5,431,940, and Pflaumer, U.S. Pat. No. 5,641,532, both of which emphasize the preparation of non-carbonated juice beverages. All three of these patents are incorporated herein by reference.

Long chain polyphosphates are effective in inhibiting the growth of microorganisms in non-carbonated beverages when present in the beverage at about 100 ppm to about 3000 ppm, typically about 300 ppm to 3000 ppm, preferably about 500 ppm to about 1500 ppm, and more preferably about 1000 ppm.

Beverages can be prepared by making a beverage concentrate, adding to it a sugar syrup containing the polyphosphate of this invention, including the acidulants, preservatives, and water in amounts sufficient to obtain the desired beverage composition. The concentrate is prepared by admixing a dispersion of the flavor oil in ester gum and a dispersion of a hydrocolloid such as gum acacia in water, then homogenizing to form a stable emulsion of the flavor oil. Aside from providing the desired flavor, this emulsion also serves to add opacity and texture to the beverage. Other suitable oils may be added to the concentrate to increase

opacity. Preservatives, acidulants, and coloring material may also be added to the concentrate. The sugar syrup is prepared separately by mixing sugar syrup such as high fructose corn syrup with water, then adding to it any optional water-soluble vitamins, the polyphosphate, acidulants, and preservatives. The sugar syrup and concentrate are combined to form a non-carbonated beverage. It can then be packaged and stored.

Non-carbonated beverages may comprise 0.1 to 40%, preferably 1 to 20%, and more preferably 2 to 10%, and most preferably 3 to 6% fruit juice (weight percentage based on single strength 1-16° Brix fruit juice). The juice may be any citrus juice, non-citrus juice, or a mixture thereof, which is known for use in non-carbonated beverage. Non-fruit juices, such as vegetable or botanical juices, and tea solids also can be used in non-carbonated beverages. When tea solids are used, the non-carbonated beverage typically comprises preferably about 0.02% to about 0.25%, more preferably about 0.07% to about 0.15%, by weight of tea solids. The term "tea solids" as used herein means solids extracted from tea materials including those materials obtained from the genus *Camellia* including *C. sinensis* and *C. assaimica.*

Non-carbonated beverages have a pH of from about 2.5 to about 4.5, preferably from about 2.7 to about 4.0. This pH range is typical for non-carbonated dilute juice beverage products. Beverage acidity can be adjusted to and maintained within the requisite range by known and conventional methods, *e.g.*, the use of food grade acid buffers. Typically, beverage acidity is a balance between maximum acidity for microbial inhibition and optimum acidity for the desired beverage flavor and sourness impression.

Non-carbonated beverages typically contain an artificial or natural, caloric or noncaloric, sweetener. Preferred are carbohydrate sweeteners, more preferably mono- and or disaccharide sugars, such as maltose, sucrose, glucose, fructose, invert sugars and mixtures thereof. The non-carbonated beverage products typically comprise from about 0.1% to about 20%, more preferably from about 6% to about 14%, sugar solids by weight of the beverage products. Optional artificial or noncaloric sweeteners include, for example, saccharin, cyclamates, acetosulfam, L-aspartyl-L-phenylalanine lower alkyl ester sweeteners (*e.g.*, aspartame), and the like.

Non-carbonated beverages may also comprise any other ingredient or ingredients typically used as optional beverage ingredients, such as flavorants, preservatives (*e.g.*, organic acids), colorants and so forth. Preservatives commonly used in beverage products include, for example, sorbates, benzoates, organic acids, and combinations thereof. Preferred preservatives are sorbic acid, benzoic acid, alkali metal salts thereof, and mixtures thereof.

Typically about 400 ppm to about 1000 ppm, more typically about 650 ppm to about 750 ppm, of preservative is added to the beverage, but use of the antimicrobial long chain polyphosphate of this invention in the beverage will allow this amount to be reduced.

Flavorants and colorants include those typically used in beverages. Typically, the colorant and flavorant are matched to produce a particular impression (*i.e.*, lime-flavored beverages are green, strawberry-flavored beverages are red; etc.). The amount added will depend on the colorant and flavorant added and the desired color and flavor of the final beverage.

Non-carbonated beverages may contain up to 110% of the U.S. Recommended Daily Allowance (RDA) of vitamins and minerals, provided that such vitamins and minerals are chemically and physically compatible with the essential elements of the non-carbonated beverage products and not cause the growth of microorganisms. Preferred are vitamin A, provitamins thereof (*e.g.*, β-carotene), and ascorbic acid. However, calcium, iron and magnesium fortification should be avoided since these polyvalent cations can bind to and inactive the polyphosphates. Gums, emulsifiers and oils, such as guar gum, xanthan, alginates, mono- and di-glycerides, lecithin, pectin, pulp, cottonseed oil, vegetable oil, food starches, and weighting oils/agents, as well as esters and other flavor and essence oils may also be included.

The balance of the beverage is added water. Added water does not include the water incidentally included in the beverage by other added materials, such as fruit juice or sugar syrup. Although the long chain polyphosphate of this invention can be used with added water of any hardness typically used to prepare beverages, the added water typically comprises 0 to about 180 ppm hardness.

INDUSTRIAL APPLICABILITY

The long chain polyphosphates, which are effective against yeast and mold growth, can help extend the shelf life of the beverage. The level of preservatives added, such as benzoates and sorbates, can be reduced, minimizing or eliminating the undesirable flavor associated with high levels of these materials. It is also possible to decrease the heat treatment, thus preserving the flavor of the beverage.

In addition, the long chain polyphosphates can be used to control yeast and mold growth in any food application in which phosphates are normally used. These applications include, for example: meat, poultry, and seafood products; imitation dairy products, such as non-dairy creamers, whipped toppings, and frozen desserts; dairy products, such as milk,

cream, cheese, whey, and ice cream; egg whites; dried and canned fruit; jellies and jams; and gums, such as carrageenan.

The advantageous properties of this invention can be observed by reference to the following examples, which illustrate, but do not limit, the invention.

Throughout this specification and claims, all parts and percentages are by weight unless otherwise indicated.

EXAMPLES

This example shows the effect of long chain polyphosphates on microorganisms.

Experimental Procedure

The juice drink was prepared to contain a minimum of five gradually decreasing levels of the test compound. Positive control samples of each product were also prepared without the test compound. Five replicates were prepared for each sample. Prior to use for juice drink preparation, the water used was adjusted to 80 ppm hardness with calcium chloride.

Inoculum

Three mixed inocula were prepared from: yeast, *Saccharomyces cerevisiae* from beer, *Rhodotorula spp.* from juice, *Candida magnoliae* from pineapple concentrate, and preservative- resistant *Zygosaccharomyces bailli*; mold, *Aspergillus niger*, *Penicillum spp.* from pineapple juice, and *Fusarium spp.* from raspberry juice; and lactic acid bacteria, *Lactobacillus plantarum* and *Lactobacillus spp.* from spoiled tomato paste. Each organism was grown separately in its appropriate medium before centrifuging, washing and combining in approximately equal numbers.

Inoculation

Following adjustment of the inoculum to the appropriate level, a minimum volume of inoculum was added to each prepared sample. Samples were inoculated to approximately 100,000 colony forming units/mL with the appropriate spoilage organism cocktail.

Incubation

Samples inoculated with yeast or mold were incubated at 25°C for five days. Samples inoculated with lactic acid bacteria were incubated at 35°C for four days.

Sampling

Following the incubation period, samples were examined for turbidity, sediment, or other visible growth as compared to the positive control.

Data Analysis

The minimum inhibitory concentration of each test compound was considered to be the lowest concentration of antimicrobial agent which resulted in complete inhibition of visible growth of the inoculated organisms during the incubation period tested.

Results

Minimum inhibitory concentrations (the minimum concentration that inhibits growth of the microorganism) for various additives were measured in an orange drink formulation at pH 3.8 at room temperature. Results are shown in Table 1.

Table 1

Additive	Minimum Inhibitory Concentration (ppm)	
	Yeast	Mold
Sodium polyphosphate (n=13)[a]	1000	1000
Sodium polyphosphate (n=21)	625	1000
Sodium polyphosphate (n=28)	625	875
Sodium sorbate	750	<250
Sodium benzoate	750	<250

[a]Commercially available food grade amorphous, water soluble sodium polyphosphate composed of linear chains of metaphosphate units with an average chain length of 13.

Sodium polyphosphates in which n is 21 or greater were more effective than sorbate or benzoate in inhibiting the growth of yeast. When higher polyphosphates are used, lower minimum inhibitory concentrations are observed. Use of polyphosphates in which n is greater than about 45 is advantageous. Use of polyphosphates in which n is greater than about 75 is especially advantageous.

The lactic acid bacteria tested did not grow in the orange juice drink, not even in the positive control. However, all yeast and mold positive controls were positive within the incubation time. Complete test results are shown in Table 2. In Table 2, "NT" means not tested

Table 2

Test Compound	Concentration (ppm)	Yeast # Positive/# Tested	Mold # Positive/# Tested
Sodium polyphosphate (n=21) **Sodium polyphosphate (n=21)**	2000	0/5	0/5
	1000	0/5	0/5
	875	NT	5/5
	750	0/5	5/5
	625	0/5	NT
	500	5/5	5/5
	250	5/5	NT
Sodium polyphosphate (n=13) **Sodium polyphosphate (n=13)**	2000	0/5	0/5
	1000	0/5	0/4
	875	NT	5/5
	750	5/5	6/6
	625	5/5	NT
	500	5/5	2/2
	250	NT	NT
Sodium polyphosphate (n=28) **Sodium polyphosphate (n=28)**	2000	0/5	0/5
	1000	0/5	0/4
	875	NT	0/5
	750	0/5	5/6
	625	0/5	5/5
	500	5/5	5/5
	250	5/5	NT
Sorbate	2000	0/5	0/5
	1000	0/5	0/4
	750	0/5	0/1
	625	5/5	0/1
	500	5/5	0/5
	250	5/5	0/5
Benzoate	2000	0/5	0/5
	1000	0/3	0/4
	750	0/5	0/1
	625	5/5	0/1
	500	5/5	0/5
	250	5/5	0/5

Having described the invention, we now claim the following and their equivalents.

CLAIMS

What is claimed is:

1. A non-carbonated beverage comprising:

0.1% to 40%, by weight, fruit juice;

about 400 ppm to about 1000 ppm of preservative; sweetener;

added water; and

about 100 ppm to about 3000 ppm of a polyphosphate;

in which:

the polyphosphate is a long chain polyphosphate in which the average number of phosphate units is greater than 22;

the beverage has a pH of about 2.5 to 4.0.

2. A non-carbonated beverage comprising:

0.02 to about 0.15%, by weight of tea solids;

about 400 ppm to about 1000 ppm of preservative; sweetener;

added water; and

about 100 ppm to about 3000 ppm of a polyphosphate;

in which:

the polyphosphate is a long chain polyphosphate in which the average number of phosphate units is greater than 22;

the beverage has a pH of about 2.5 to 4.0.

3. A non-carbonated beverage comprising:

flavorant;

colorant;

about 400 ppm to about 1000 ppm of preservative; sweetener;

added water; and

about 100 ppm to about 3000 ppm of a polyphosphate;

in which:

the polyphosphate is a long chain polyphosphate in which the average number of phosphate units is greater than 22;

the beverage has a pH of about 2.5 to 4.0.

4. A the non-carbonated beverage of claim 3 additionally comprising 0.1% to 40%, by weight, of fruit juice.

INTERNATIONAL SEARCH REPORT

Int. ional Application No
PCT/US 00/17548

A. CLASSIFICATION OF SUBJECT MATTER
IPC 7 A23L2/44 A23F3/16 A23L3/3553 A23L2/02

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
IPC 7 A23L A23F

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)
EPO-Internal, WPI Data, PAJ, FSTA

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category °	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	WO 95 22910 A (PROCTER & GAMBLE) 31 August 1995 (1995-08-31) cited in the application claims 1,3,7,8,10; example 1 page 3, line 30 -page 4, line 16 page 5, line 16 -page 6, line 8 page 12, line 7-10 page 13, line 6-9 ---	1-4
X	WO 97 21359 A (PROCTER & GAMBLE) 19 June 1997 (1997-06-19) page 3, line 4-20; claims 1-3; examples 1,2 page 4, line 1,2 page 6, line 1-4-22 page 8, line 3-33 page 12, line 1-9 --- -/--	1-4

[X] Further documents are listed in the continuation of box C.

[X] Patent family members are listed in annex.

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"A" document defining the general state of the art which is not considered to be of particular relevance

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Date of the actual completion of the international search	Date of mailing of the international search report
19 October 2000	26/10/2000

Name and mailing address of the ISA	Authorized officer
European Patent Office, P.B. 5818 Patentlaan 2 NL – 2280 HV Rijswijk Tel. (+31–70) 340–2040, Tx. 31 651 epo nl, Fax: (+31–70) 340–3016	Tallgren, A

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INTERNATIONAL SEARCH REPORT

Int[illegible]ional Application No
PCT/US 00/17548

C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

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